Laboratory Stakeholders Workgroup

Summary of May 12, June 16, July 14, and August 18, September 29 and December 8, 2008 Meetings

Attendance: Organizations represented were Virginia Municipal Wastewater Association, Virginia Manufacturing Association, Virginia Rural Water Association, Virginia Water Environment Association Laboratory Practices Committee, commercial laboratories, and DEQ.

The following laboratory related recommendations were agreed upon by members of the workgroup. DEQ will consider these recommendations when developing further guidance.

TOPICS

1. ***Standard Methods*** **(SM) Part 1000**

   VPDES permits require proper operation and maintenance which includes "adequate laboratory controls and appropriate quality assurance procedures." The work group agreed that following SM Part 1020, with modifications, along with QC components recommended by EPA would fulfill the permit QA/QC requirements for compliance analyses. The components given below are the minimum QC to be applied to all testing procedures where applicable, not just SM methods. A laboratory is to follow the frequency and acceptance criteria given in the approved edition of SM cited by the laboratory.

   a. Initial Demonstration of Capability (IDC)
      - A facility/laboratory training program that is accepted by DEQ may be used as an alternative to analyzing four replicates of an independent check sample. This will allow facilities that already have an extensive well documented training program in place to continue using them without incurring additional costs.
      - Components of an alternate training program to be presented to DEQ must include the following:

        Demonstrated knowledge of the method

        Demonstrated ability to conduct all aspects of the method the analyst will perform

        Analysis of at least two replicates of an independently prepared check sample

   b. Matrix spikes
      - Matrix is defined as wastewater.
      - Captive labs are expected to rotate outfalls when selecting samples for spiking.
      - Commercial labs are to randomly select samples for spiking. When a client requests that a specific sample be spiked, this sample may occasionally be used in place of a randomly selected sample;

c. Analysis of externally supplied standards
-Recovery must meet method/SM/DEQ established acceptance criterion.
- DEQ's established criteria of 80 -120% recovery is to be used in place of control charts when a method requires the laboratory to establish the acceptable limits.
- Manufacturers' established acceptance criteria may not be used because they are parameter specific, not method specific.

d. Analysis of reagent blanks
-Glassware used in the analysis of blanks must be randomly selected, unless specified otherwise in the method.

e. Calibrations with standards – no modifications were suggested.

f. Analysis of analytical duplicates
Analytical duplicates should be split from the sample container received by the laboratory.

Field measurements, defined as "required analysis within 15 minutes of collection" (i.e., pH, DO, TRC, and temperature), will not require analytical duplicates. Removal of this requirement for these parameters is based on problems associated with reporting min/max limits in permits and the possibility of a rapidly changing concentration in a given sample.

g. Positive and negative controls for microbiological pollutants
-Positive control may be a dilute influent from a source within the wastewater treatment process containing the microorganism(s) of interest.

-Negative control may be a sterile-water blank for laboratories using manufacturer-prepared media; laboratories preparing their own media (i.e., using media other than unit-dose-media such as Colilert) must also inoculate the prepared media with a culture known to produce a negative response.

h. Corrective action for failed QC
-Laboratory/facility must appropriately address failed QC and provide adequate documentation of corrective actions taken.

2. **Ortho-phosphate requirement in Watershed Nutrient Trading General Permit**

The Chesapeake Bay Watershed Nutrient Trading General Permit requires monitoring of ortho-phosphate using approved methods and holding times given in 40 CFR Part 136. This data is required in the general permit for the Chesapeake Bay Program modeling. For several years prior to the issuance of the general permit, some permittees from Maryland, Virginia, Pennsylvania, and DC have voluntarily submitted phosphorus results to the Bay Program for samples that were either filtered or not filtered.

DEQ Chesapeake Bay Program has indicated that there is no EPA guidance identifying the type of reactive phosphorus analysis that should be performed (filtered or unfiltered). The Watershed Nutrient Trading General Permit requires ortho-phosphorus monitoring and filtering of the sample within 15 minutes of sample collection. Since either filtered or unfiltered are acceptable for the Chesapeake Bay program modeling, DEQ Inspection Program will be provided guidance to also accept unfiltered sample as meeting this general permit requirement.

3. **Thermistor temperature calibration**

Verification of thermistors used in pH meters and dissolved oxygen meters for automatic temperature correction (ATC) was discussed. The Work Group agreed that the Virginia Environmental Accreditation Program (1VAC30-45-860 and 1VAC30-46-210) requirements for annual calibration/verification annual over the entire range of use should be utilized. For a thermistor or thermometer that is used to measure one target temperature a one point calibration at the target temperature meets this requirement.

4. **Field duplicates vs. lab duplicates**

See Section 1.f . above.

5. **QL's listed in permits**

When developing limitations for VPDES permits DEQ utilizes site specific discharge and receiving stream information to evaluate if there is a potential to violate water quality criteria. To determine if permit limitations are required DEQ identifies site specific target values (SSTV) for parameters of concern associated with the individual discharge. These SSTVs are used to establish the QLs for parameters of concern required as part of the VPDES application for reissuance.

The work group reviewed "worst case" (hardness, pH, stream flow) SSTV scenarios for number of parameters. As listed below there are a number of parameters for which there is not an achievable QL under the "worst case" scenario for a site specific target value.

Parameter	Worst Case for Site Specific Target Value (ug/l)	Achievable QL
Antimony	1.4	YES
Arsenic	1.0	YES
Cadmium	0.057	YES
Chromium III	3.6	YES
Chromium VI	1.6	NO
Copper	0.36	NO
Lead	0.35	YES
Mercury	0.005	YES
Nickel	0.94	YES
Selenium	0.75	YES
Silver	0.0032	NO
Zinc	3.6	YES

Pentachlorophenol	0.0015 (1)	NO
Hydrogen sulfide (2)	0.05 (1)	NO
Cyanide	1.3 (1)	NO

DEQ should insure that permit writers are utilizing existing guidance for requiring QLs to address specific permit requirements. This includes clarification that QLs should only be established to a level that is necessary to determine compliance with individual permit site specific target values. DEQ will continue to advise permittees to use clean sampling protocols for low level analysis.

DEQ should include stakeholder participation as part of the process to revise guidance associated with QL requirements in VPDES permits.

6. **Clarification of DEQ's definitions associated with lab work**
    - Laboratory Control Sample (LCS), Laboratory Intercomparison Samples (LIS), and Proficiency Evaluation Sample (PES) are quality control samples of known concentration prepared from a source different from the one used to prepare standards.
        - LCS is required for VPDES. It is equivalent to the laboratory fortified blank (LFB) which also is from an alternate source and may be prepared by the laboratory.
        - LIS is not required for VPDES.
        - PES is required only for permittees participating in the Discharge Monitoring Report Quality Assurance Program (DMRQA). This sample must be purchased from an approved PT Provider. The concentration must not be known by the laboratory or permittee.
    - Annual – once per calendar year

- Quarterly –once within the calendar quarter
- BOD5 – final reading must be taken on the fifth day of incubation unless the edition of Standard Methods used by the laboratory establishes a more stringent requirement. (Previously the lab was instructed to take the final reading within plus or minus three hours of the initial reading.)
- Externally Supplied Standard – sample of known concentration prepared using a different source than was used to prepare the calibration standards.

7. DEQ Inspection Consistency:

DEQ need to have a process for providing changes to the program to DEQ Inspectors and the regulated community so that there is a clear understanding of the new requirements and that DEQ consistently applies the requirements throughout the state. (example: calibration requirements for thermistor or thermometer)

DEQ should ensure that present guidance requiring a final inspection response letter from DEQ indicating that deficiencies noted in the inspection "have been addressed" is being followed.

Before sending an inspection report DEQ should call the inspected facility/laboratory to clarify report exceptions (to extent possible) identified during review of the facility/laboratory records that were not discussed during the closing interview at the time of the inspection. This approach may allow for clarification and verification of issues prior to the report or help the facility more fully understand the issue when the inspection report is received.

DEQ should classify inspection report exceptions as "comments" or "required actions."

DEQ will examine the use of unannounced inspection to the extent possible recognizing the mandate by the General Assembly to conduct unannounced inspections.

DEQ should continue this workgroup on an annual/semiannual basis to discuss emerging issues as well as to provide a forum to stakeholders to discuss concerns.

8. Composite Requirements for VPDES Permit Application Attachment A Sampling

Certain parameters in Attachment A (base /neutral/acid extractable organic compounds, pesticides and PCBs) are currently required to be monitored via "special composite" or grab samples. The "special composite" sample was initiated by DEQ to address concerns of inaccurate results if a normal composite sample were used for those

pollutants. Based on further DEQ review, DEQ is not aware of any other states that have a similar requirement. Discussions with EPA Region III confirm that Regional III also utilizes standard composite procedures to sample for these parameters. Consequently, DEQ should no longer require the use of "special composite" samples. Attachment A should be revised to reflect the use of "standard" composite or grab samples. For existing permits with the "special composite" requirements DEQ should allow permittees to use the "standard" composite in place of the "special composite" sample.

Other points of discussion:

- Laboratory inspection check sheets and Frequently Asked Questions document will be updated to reflect QA/QC requirement modifications in Topic 1. This Work Group and the Good Laboratory Practices Committee should be provided an opportunity to comment on the draft laboratory inspection check sheets.

- Labs may establish criteria for acceptable relative percent differences (RPD) of duplicates when the concentration of analyte is too low to allow reasonable statistical evaluation.

- The Laboratory Work Group should continue to meet on a semiannual basis to discuss emerging issues.